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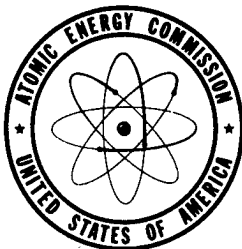
**EFFECT OF SODIUM-POTASSIUM ALLOY ON
VARIOUS MATERIALS AT ELEVATED
TEMPERATURES**

By
LeRoy R. Kelman

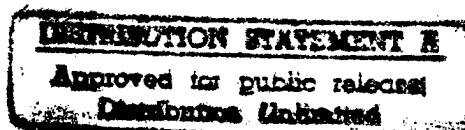
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EFFECT OF SODIUM-POTASSIUM ALLOY ON VARIOUS MATERIALS
AT ELEVATED TEMPERATURES

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December 1, 1946

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at Elevated Temperatures

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Effect of Sodium-Potassium Alloy on Various Materials
at Elevated Temperatures

A. Purpose

Interest in sodium-potassium alloys as thermo-vehicles has resulted in this investigation of the corrosion effects of these alloys on various materials at elevated temperatures. While this investigation is still in progress, it is felt that the results obtained to date might be of some use to those who are confronted with problems of handling Na-K alloys at elevated temperatures.

Tests were made at temperatures as high as 800°C. The maximum temperature of Na-K alloy contemplated for the Argonne Laboratory's fast neutron pile is 450-500°C but, if materials can be found which will withstand Na and K at their boiling temperatures (892°C and 774°C), the field of usage of Na-K alloys will be considerably broadened.

B. Summary of Conclusions

1. Uranium, thorium, beryllium, and columbium show extremely good resistance to pure Na-K alloy. The presence of oxygen in the Na-K alloy appears to effect the corrosion resistance of these metals.
2. Pure iron and low-carbon steels have good resistance to Na-K alloys and appear to be suitable metals for construction of equipment to handle Na-K where structural strength and air corrosion resistance at elevated temperatures are not necessary. The presence of chromium and high chromium alloys appears to greatly accelerate the rate of attack of steel by Na-K alloy.
3. Nickel and high nickel alloys have extremely good resistance to Na-K alloy.
4. Type 302 stainless steel (18-8) shows intergranular corrosion when tested in Na-K alloy.
5. In general, copper and its alloys, carbon, silicon, silver, and platinum are extensively attacked by Na-K alloy.

C. Equipment and Procedure

Sodium melts at 97.5°C, potassium at 63.5°C, and alloys of the two melt at lower temperatures. To facilitate ease in handling, an alloy of equal weights of sodium and potassium which is liquid at room temperature (approximate melting point 12°C) was arbitrarily chosen to be used for all corrosion tests. Materials which can be used to handle this alloy will probably be equally effective in handling other alloys of sodium and potassium. A summary of known properties of sodium, potassium, and their alloys is given in CP-3061.

Both static and dynamic tests have been made for this investigation. The equipment used for static tests is shown in Figures 1 and 2. A battery of six static corrosion units was made using nichrome wound furnaces which can be operated continuously up to 900-1000°C and controlled to $\pm 5^\circ\text{C}$. All tests were made in an argon atmosphere, and it was found advisable to equip each bomb with a pressure release which consisted of a vertical pipe extending below a mercury well.

Threaded joints do not hold hot Na-K alloy. The most satisfactory joint other than a welded joint was found to be a metal ring seal with oval cross section which seats between flanges in V-shaped grooves as shown in Figure 1 (a). This type of seal was used on bombs made of materials such as stainless steel and nickel which do not have to be protected against oxidation in air at elevated temperatures. Bombs made of low-carbon steel were designed as shown in Figure 1 (b). The low-carbon steel bomb was enclosed in a stainless steel container and both extended far enough out of the furnace so that an air-cooled neoprene gasket could be used as a seal for both bomb and container. Thermocouple wells made of the same material as the bomb extended into the Na-K alloy to where the corrosion specimens were located.

The Na-K alloy used for most of the static corrosion tests was made by melting sticks of sodium and potassium under an argon atmosphere and kept under mineral oil. For most tests, 100 cc. of Na-K were transferred to the bomb by pipetting under an argon atmosphere. The Na-K alloy now being used was obtained from Mines Safety Appliance Company and is considerably purer than the Na-K alloy made from sodium and potassium sticks.

Corrosion specimens for static tests were either cylinders 1" long and 1/2" in diameter or sheet, depending on the material available. The specimens were ground and polished, cleaned in acetone, and weighed and measured before testing. The inside surface of the bomb was polished, cleaned, and dried with acetone before each test. Specimens were removed from test by bringing them through a layer of mineral oil on the surface of the Na-K alloy in the bomb. This prevented oxidation of the Na-K alloy which adhered to the specimens, thus permitting easy inspection of the specimens before removing from the Na-K. The specimens were cleaned by immersing rapidly in water and drying immediately in acetone. The weight and dimension changes of the specimens were then noted, and the specimens were examined metallographically for type and depth of corrosion and changes in structure due to the high temperature corrosion test.

When there appeared to be appreciable solution of the material being tested, samples of the Na-K alloy were taken for chemical analysis. These samples were taken when the Na-K alloy had cooled to room temperature, and no attempts were made to determine solubilities at elevated temperatures.

Use of Na-K alloy as a coolant will entail the motion of the liquid alloy which will add mechanical erosion to the corrosion and solution of static uses. To study the resistance of materials of construction to this action and to gain experience in pumping and handling large quantities of hot Na-K alloy, a large dynamic system was made as shown in Figure 3. The entire system was made of low-carbon steel and consisted essentially of a loop of 1" I.D. steel tubing through which Na-K alloy was pumped at velocities of 2 to 3 feet/second. About 50 KW were introduced by shorting a 3 foot length of pipe across a 120 KVA transformer and heating by electrical resistance. The Na-K was cooled before returning to the pump tank by means of Dowtherm A in a tube and shell heat exchanger, and water was used to cool the Dowtherm A. Dowtherm A does not react appreciably with Na-K. As a safety precaution, water is never used against pipes carrying Na-K alloy.

In this dynamic system, the Na-K was heated to 475°C and cooled to 175°C. To prevent oxidation of the steel tubing and as a safety precaution in case of leaking of the ring seal joints, the entire system was enclosed in a sheet metal housing in which a nitrogen atmosphere was maintained. A slow flow of purified nitrogen was maintained in the pump tank so as to prevent air from entering along the vertical pump shaft.

Tubular specimens were placed in both the hot and cold portions of the system so that deposition of material from the Na-K on cooling could be followed as well as removal of metal in the hot portion of the system. A thin annulus of Na-K was maintained between the specimens and the steel tubing which served as an excellent heat transfer medium since steel is thoroughly wetted by Na-K and iron oxides are readily reduced. The hot specimens were located in the heated section of steel tubing and, therefore, were progressively hotter in the direction of Na-K flow. The temperatures of the two end specimens were recorded by thermocouples as the two extreme temperatures. However, it was found that the two specimens next to the one on the hot end were hotter than the end specimen because considerable heat was removed by the heavy bus bar terminal which was close to the end specimen.

Some work was done on a method of measuring very small corrosion rates by radioactive means, and, though not thoroughly developed, this method showed enough promise to justify a short discussion. A 1/2 mil layer of radioactive iron was plated on the inside surface of tubular Armco Iron specimens. The specimens were then heat treated in a hydrogen atmosphere to ensure reduction of oxides and good adherence of the plate. A measure of the corrosion rate was then obtained by measuring the loss in radioactivity of the specimen as a result of dynamic corrosion testing.

Dynamic corrosion systems (Figure 4) are now being made in which Na-K alloy is circulated by thermal convection. The Na-K alloy is heated near the bottom of a closed loop of pipe by means of globar heating elements in a multiple tube heater and cooled near the top of the system by means of Dowtherm A in a finned tube and shell heat exchanger. In this system, there are no pumping problems, and long time tests can be made with a minimum of maintenance. With a temperature difference between the hottest and coldest part of this system of 300°C, the rate of flow of Na-K alloy in a 1" I.D. pipe is approximately 1 to 2 feet/second.

D. Discussion of Results

Before discussing the results of the corrosion tests, emphasis should be placed upon the fact that the Na-K alloy used in most of the tests contained considerable oxygen and probably other impurities. Unless otherwise stated, this Na-K alloy was used on all tests herein reported. Na-K alloy now being used was obtained from the Mines Safety Appliances Company, and tests of metals which are good getters indicate that this alloy is considerably purer than the alloy previously used. The Dow Chemical Company has reported finding large quantities of hydrogen in the Na-K alloy they have been using. Methods for determining oxygen and hydrogen in Na-K alloy are now being developed by our Chemical Division.

The materials tested and their approximate chemical compositions are given in Table I. The test results are summarized in Table II.

1. Uranium and Thorium

A German report⁽¹⁾ described alloys of thorium and sodium. However, several attempts were made to encourage uranium and thorium to react with sodium and Na-K alloy with no success. Only a small trace of uranium or thorium could be found in the Na-K alloy after these tests. Samples for chemical analysis were taken from near the top of the Na-K to avoid including uranium and thorium oxides which accumulated at the bottom of the test bomb. The weight changes of the specimens depended on the oxygen content of the Na-K alloy. When the Na-K alloy contained considerable oxygen, the uranium or thorium specimens were coated with heavy oxide layers which tended to flake off during test, resulting in large weight losses. See Figures 5, 6, and 7. The rate of weight loss at temperatures from 600°C to 800°C for 1 to 3 day tests was on the order of

-1 to -5 mg/cm²/hour for uranium and
-0.1 to -3 mg/cm²/hour for thorium.

When M.S.A. Na-K alloy which had been deoxidized with uranium turnings was used, the uranium or thorium specimens were coated with thin tenacious oxide films and gained weight. The rate of weight gain after 6 days at 600°C was only

+0.009 mg/cm²/hour for uranium and
+0.006 mg/cm²/hour for thorium.

2. Beryllium

The rate of attack of beryllium by Na-K alloy is very small. A specimen was tested under static conditions for 3-1/2 days at 800°C. When removed from test, it was covered with a very light grey material which did not adhere to the specimen. Under this film, the specimen was bright and appeared

(1) "The Alloys of Thorium with Copper, Aluminum and Sodium", Von G. Grube and L. Botzenhardt; Ztschr. Elektrochem. Bd. 48, Nr. 8, 1942.

to be etched. Testing of the same specimen was continued under similar conditions for 30 days, and the rate of weight loss decreased from

-0.05 mg/cm²/hour for 3-1/2 days to
-0.005 mg/cm²/hour for 30 days.

Although the specimen surface appeared to show deep intergranular corrosion, metallographic inspection revealed only limited attack as shown in Figures 8 and 9.

When the relatively pure M.S.A. Na-K alloy was received, qualitative tests were made to determine the effect of oxygen in Na-K on beryllium. One specimen was tested in M.S.A. Na-K which had been deoxidized with uranium turnings, and another specimen was tested in N.S.A. Na-K to which considerable oxygen had been added. After 6 days at 600°C the rates of weight changes were

-0.0015 mg/cm²/hour for the deoxide Na-K and
+0.003 mg/cm²/hour for the oxidized Na-K.

The first specimen had an extremely thin gray film, whereas the latter specimen was covered with a similar grey film of appreciably greater thickness.

Nine beryllium specimens were tested in the dynamic system. After 6 days, the specimens near the cold end of the heater tube remained bright and gained up to

+0.0034 mg/cm²/hour at approximately 200-300°C.

The specimens became progressively darker with increasing temperature until they had a black film at the hot end and, when weighed with the film intact, lost

-0.0124 mg/cm²/hour at approximately 500°C.

The relatively large rate of weight loss at approximately 500°C, when compared with static tests at 600°C, indicates that the film that forms on beryllium is removed by the moving Na-K. It should be noted that, though the Na-K used in this test was M.S.A. Na-K, it had been used for several dynamic tests of iron and probably was contaminated with considerable oxygen and other impurities.

3. Columbium

Columbium was only lightly attacked by the Na-K alloy. A rough test in impure Na-K alloy at 800°C indicated that columbium was considerably attacked by Na-K. However, when tested in deoxidized M.S.A. Na-K for 6 days at 600°C, the rate of weight loss was only

-0.0016 mg/cm²/hour in a low carbon steel bomb as compared to
-0.0064 mg/cm²/hour in an 18-8 stainless bomb.

The first specimen had a dull grey surface whereas the latter had a black non-adherent film which has not been identified.

4. Iron and Low-Carbon Steel

Armco iron and low-carbon steel, when tested alone, have extremely good resistance to corrosion by Na-K alloy and appear to be suitable metals for construction of Na-K handling equipment where structural strength and air corrosion resistance at elevated temperatures are not necessary. Any iron oxide on the specimens is rapidly reduced by the Na-K, and the specimen surfaces are thoroughly wetted and remain clean and bright, thus resulting in ideal conditions for efficient heat transfer. While the corrosion rate of iron and low-carbon steel is small, it was considered important enough to investigate thoroughly.

Attempts were made in several static tests to determine the rate of attack of S.A.E. 1020 steel as a function of temperature, time, and the presence of other metals. Also, several dynamic tests were made on Armco iron, plated radioactive iron, and S.A.E. 1020 steel. In most cases the weight losses were so small that experimental errors confused the results. However, some definite trends were noted. Several points indicate that the mechanism of corrosion of low-carbon steel consists mainly of removal of carbon rather than iron and that the rate of corrosion is related to the mode of occurrence of carbon. In dynamic tests for 6-1/2 and 13 days at approximately 500°C, S.A.E. 1020 steel showed up to 20 mils surface decarburization and extensive grain growth in the decarburized areas as shown in Figures 10 and 11. The rate of weight loss for these tests was on the order of

-0.001 mg/cm²/hour.

In similar dynamic tests, Armco iron and pure plated iron lost

-0.0001 and -0.0007 mg/cm²/hour.

The greatest rate of weight loss of S.A.E. 1020 steel when tested alone under static conditions was on the order of

-0.01 to -0.02 mg/cm²/hour at 700°C

as compared to

-0.001 to -0.002 mg/cm²/hour at 800°C.

Figures 12, 13, and 14 show the effects of tests at 700°C and 800°C on the structure of the steel. The test at 700°C, which is slightly below the eutectoid temperature, resulted in extensive spheroidization of carbides throughout the specimen and total surface decarburization up to a depth of 10 mils with partial decarburization of 25 mils. The test at 800°C, which is above the eutectoid temperature, resulted in recrystallization and general grain coarsening with practically no surface decarburization. The greater corrosion rate at the lower temperature was verified by several check tests. This anomaly seems to be due to the presence of carbon as the carbide at 700°C whereas most of it is dissolved in gamma iron at 800°C.

Although the data obtained were not accurate enough to draw a definite curve, tests of S.A.E. 1020 steel for various lengths of time in the dynamic tests and in static tests at 700°C and 800°C indicated that the rate of weight loss decreases with time. The rate of weight loss at 700°C was

-0.02 mg/cm²/hour for 1 and 2 days

as compared to

-0.01 mg/cm²/hour for 7 days.

The rate of weight loss at 800°C was

-0.002 mg/cm²/hour for 8 days

as compared to

-0.001 mg/cm²/hour for 30 days.

Tests of S.A.E. 1020 steel in the presence of several metals indicate that, when chromium is present, the rate of attack of the steel is sometimes greatly accelerated. Separate static tests at 800°C of pure chromium, 18-8 austenitic stainless steel, and 17% chromium ferritic stainless steel in low-carbon steel bombs resulted, in each case, in deposition of iron crystals on the bomb wall at the Na-K surface as shown in Figure 15. However, in one case it was found that the testing in a low-carbon steel bomb of an alloy containing a large amount of chromium did not result in deposition of iron crystals. This alloy was Inconel which contains 79.5% Ni, 13% Cr, and 6.5% Fe. In a 7-1/2 day test in the presence of 18-8 stainless steel, S.A.E. 1020 steel lost weight at the rate of

-0.08 mg/cm²/hour at 800°C.

However, the rate of weight loss in a similar test for 9-1/2 days was only

-0.0001 mg/cm²/hour at 400°C.

These results suggest that we must be cautious about using other metals in the same system with iron especially at elevated temperatures.

Samples of the Na-K alloy from several of the static tests were checked spectroscopically for iron content. In no case was there greater than 20 ppm of iron which is approximately the iron content of the Na-K before the test. The solubility of iron at elevated temperatures was not investigated.

5. Nickel and Its Alloys

Nickel and its alloys were found to have extremely good resistance to corrosion by Na-K alloy. Commercially pure nickel, when tested in

18-8 stainless steel bombs at 600°C, lost only

-0.0005 mg/cm²/hour in 1 day and
-0.0001 mg/cm²/hour in 6 days.

When tested in low-carbon steel bombs, commercially pure nickel and all alloys containing high nickel content form a very thin dull-grey film (see Figures 16 and 17) and gain slightly in weight. There is considerable evidence to show that this film is a solid solution of iron and nickel. Commercially pure nickel, when tested under static conditions in a low-carbon steel bomb for 5 days at 600°C gained

+0.003 mg/cm²/hour.

When tested in pure M.S.A. Na-K for 6 days at 600°C, it gained only

+0.0007 mg/cm²/hour.

In a dynamic test for 6 days at temperatures up to 500°C in an all plain-carbon steel system, nickel specimens had a dull appearance, but the weight change was negligible.

A specimen of Z-nickel, when tested for 7 days at 600°C in a low-carbon steel bomb, was covered with a dark film and gained

+0.02 mg/cm²/hour.

Iron-nickel alloys of the Invar type containing approximately 35% nickel also had extremely good resistance to attack by Na-K alloy. There was no appreciable change in the appearance of Invar when tested in an 18-8 stainless steel bomb and in a low-carbon steel bomb at 600°C for 6 days, and the specimens gained only

+0.0006 and +0.0007 mg/cm²/hour.

When tested in a low-carbon steel bomb at 800°C for 6 days, Invar gained

+0.005 mg/cm²/hour.

Metallographic inspection of the Invar specimens revealed recrystallization of the original rolled structure at 600°C and extensive grain growth at 800°C as shown in Figures 18, 19, and 20. Attack by the Na-K alloy consisted of incipient intergranular corrosion.

Inconel, which contains 79.5% Ni, 13% Cr, and 6.5% Fe, was another high nickel alloy which withstood corrosion of Na-K alloy especially at low temperatures. A 30-day test at 300°C in a low-carbon steel bomb did not change the appearance or weight of an Inconel specimen. When tested at 600°C for 6 days in an 18-8 stainless steel bomb, Inconel was covered with a grey film having a greenish tinge and gained weight at the rate of

+0.003 mg/cm²/hour.

However, when tested in low-carbon steel bombs at 600°C and 800°C, Inconel took on the light grey coating characteristic of high nickel alloys when tested with iron, and the specimens showed appreciable weight increases. The rates of increase were

+0.01 mg/cm²/hour at 600°C for 5 days
+0.1 mg/cm²/hour at 800°C for 6 days.

Metallographic inspection of the Inconel specimens (Figures 21, 22, and 23) revealed no appreciable grain growth at 600°C. However, at 800°C the structure became unstable and particles precipitated and agglomerated into spheroids in the base metal. Also, there was intergranular attack resulting in a fingered layer of chromium oxide, and on this was deposited a continuous layer of the iron-nickel solid solution.

Monel metal was also unaffected by Na-K alloy when tested in a low-carbon steel bomb at 300°C for 30 days. When tested at 600°C for 6 days in an 18-8 stainless steel bomb, Monel was lightly etched and lost

-0.001 mg/cm²/hour.

However, here again a test in a low-carbon steel bomb at 600°C for 7 days resulted in the characteristic grey film and the specimen gained

+0.02 mg/cm²/hour.

In this case, the film, which was 0.5 mils thick, was easily stripped from the specimen. A chemical analysis of the film and the original material gave the following results:

	<u>Specimen</u>	<u>Film</u>
Nickel	65.3%	29.4%
Copper	28.4	5.7
Iron	0.37	61.0

The film is seen to consist mainly of iron and nickel in about a two to one ratio, and it is apparent that the iron must have come from the low-carbon steel bomb.

An alloy containing 80% Ni and 20% Mn was tested in Na-K alloy in an 18-8 stainless steel bomb. After 5 days at 600°C, it had a bright appearance and gained

+0.002 mg/cm²/hour.

6. Chromium and Its Alloys

As was mentioned under the discussion of high temperature tests of low-carbon steel, the presence of pure chromium or alloys containing large amounts of chromium sometimes greatly accelerates the attack of low-carbon steel by Na-K alloy. In fact, if the test is made in a steel bomb or if there is considerable surface of iron exposed to the Na-K, iron crystals are deposited on the bomb wall at the Na-K surface. See Figure 15.

Pure chromium was tested in a low-carbon steel bomb at 800°C for 10 days to determine whether or not it was the element responsible for the increased rate of attack of the steel. The chromium used was in the form of small pieces which made the determination of weight changes very difficult. However, iron crystals were deposited as described above.

Type 430 ferritic stainless steel containing 17% chromium, when tested in a low-carbon steel bomb at 800°C for 7 days, had a green chromium oxide coating and lost

-0.07 mg/cm²/hour.

The loss in weight can be attributed to the nature of the oxide which was powdery and easily removed.

Type 302 austenitic stainless steel containing 18% chromium and 8% nickel, when tested in a low-carbon steel bomb at 400°C for 9-1/2 days, had a dull grey appearance and gained only

+0.0008 mg/cm²/hour.

At this temperature, attack of low-carbon steel by Na-K alloy was not accelerated by the presence of 18-8 stainless steel. However, at 800°C for 7-1/2 days, acceleration of attack of low-carbon steel did occur, and the 18-8 stainless steel had a dull grey film and gained

+0.06 mg/cm²/hour.

Metallographic examination of the 18-8 stainless steels tested revealed incipient intergranular attack as a result of the 400°C test and very extensive intergranular attack forming chromium oxide as a result of the 800°C. See Figures 24 through 30. These specimens were covered with the same type of iron-nickel film found on nickel alloys when tested in the presence of iron. It was found that sufficient chromium had been removed from solid solution in the non-magnetic austenite near the surface of the specimen to transform that portion of the specimen to magnetic ferrite. From these results, it is apparent that oxygen in the Na-K alloy is the cause of intergranular attack of ordinary 18-8 stainless steel. The standard columbium or molybdenum stabilized stainless steels (types 347 and 316) may eliminate this problem.

7. Copper and Its Alloys

Pure copper has appreciable solubility in Na-K alloys at elevated temperatures. At 800°C for 4 days copper lost

-2.5 mg/cm²/hour,

and copper crystals were found on the walls of the low-carbon steel bomb. The Na-K alloy contained less than 10 ppm. of copper after the test.

A 9% aluminum bronze was found to have the best resistance to Na-K alloy of all the high copper alloys tested. When tested at 800°C for 3

days in a low-carbon steel bomb, aluminum bronze lost

-0.015 mg/cm²/hour.

A 10% tin bronze was badly attacked when tested at 700°C for 8 days. The tin phase was removed and the rate of weight loss was

-0.9 mg/cm²/hour.

A 5 mil sheet of 70% Cu-30% Mn alloy was completely dissolved in Na-K at 600°C for 6 days and copper colored crystals were found on the bomb walls.

8. Carbon and Cast Iron

Na-K alloy attacks carbon in an interesting manner. When a solid piece of graphite weighing 5 grams was placed in 10 grams of Na-K alloy at room temperature and allowed to stand over night, the graphite sample was entirely broken up, and minute particles were dispersed throughout the Na-K, resulting in a thick sludge. Considerable work has been done by our Chemistry Department to determine the solubility of carbon in Na-K alloy. They have not found any appreciable solubility, but they have observed swelling of graphite to about three times its original size and then thorough breaking up and dispersion of the graphite. This is believed to be due to splitting apart of atomic layers of carbon.

Because of this action of Na-K alloy on graphite, materials containing graphite cannot be used where they might come in contact with Na-K alloy. For example, cast iron, when tested in Na-K alloy, swelled and distorted badly and was found to be impregnated with Na-K alloy which replaced the graphite flakes.

9. Others

A 17 gram sample of silicon was completely dissolved when tested at 800°C for 6 days. The silicon alloyed with the low-carbon steel bomb forming a thick brittle shell containing 12% silicon, as shown in Figures 31 and 32. At room temperature the Na-K alloy contained less than 50 ppm. of silicon.

A 10 gram sample of silver was completely dissolved when tested at 800°C for 1-1/2 days. As the Na-K cooled, the silver precipitated out of solution in the form of small flakes which settled to the bottom of the bomb. At room temperature the Na-K alloy contained less than 10 ppm. of silver.

A sample of platinum was suspended by a nickel wire in a nickel bomb so that the platinum did not touch the bomb. The specimen dissolved when tested at 600°C for 1 day, but not even a trace of platinum could be found in the Na-K alloy. All of the platinum was found in a thin layer on the inside surface of the nickel bomb. Platinum apparently has appreciable solubility in Na-K alloy at 600°C.

F. Conclusions

1. Oxygen in Na-K alloy appears to effect corrosion of materials by Na-K.
2. Neither thorium nor uranium could be made to alloy with sodium or Na-K alloy. This contradicts a German report describing alloys of thorium and sodium.
3. Uranium and thorium react with oxygen in Na-K alloy at elevated temperatures to form heavy oxide layers which spall off during test. In deoxidized M.S.A. Na-K, uranium and thorium take on thin oxide layers but are otherwise not effected.
4. Beryllium is only slightly attacked by Na-K alloy at temperatures as high as 800°C. There is indication that the thin film formed on beryllium is removed in moving Na-K which results in appreciable weight losses.
5. Columbium is only slightly attacked by Na-K alloy at 600°C.
6. Iron and low-carbon steel bombs, when tested alone, have good resistance to attack by Na-K alloy and appear to be suitable metals for construction of Na-K handling equipment where structural strength and air corrosion resistance at elevated temperatures are not necessary. Thorough wetting of steel surfaces by Na-K result in ideal conditions for efficient heat transfer.
7. The greater rate of weight loss of S.A.E. 1020 steel at 700°C than at 800°C is attributed to decarburization at 700°C where carbon is present as carbide and lack of decarburization at 800°C where carbon is dissolved in gamma iron.
8. Preliminary tests indicate that the rate of weight loss of S.A.E. 1020 steel decreases with time and seems to be related to the extent of surface decarburization.
9. Tests of S.A.E. 1020 steel in the presence of several metals indicate that, when chromium or high chromium alloys are present, the rate of attack of the steel is greatly accelerated. Tests of pure chromium, 18-8 stainless steel, and 17% chromium stainless steel in low-carbon steel bombs resulted in deposition of iron crystals at the Na-K surface.
10. Nickel, and high nickel alloys such as Invar, Inconel, Monel, and 80% Ni-20% Mn have good resistance to Na-K alloys. When tested in the presence of iron, nickel alloys acquire a thin surface layer of iron-nickel solid solution.
11. Type 302 stainless steel (18-8) showed intergranular corrosion when tested in Na-K alloy. Standard columbium or molybdenum stabilized stainless steels (types 347 and 316) should resist this type of attack.
12. Copper, 10% tin bronze, and 70% Cu-30% Mn have poor resistance to Na-K alloy.

13. A 9% aluminum bronze showed fairly good resistance to Na-K alloy at temperatures as high as 800°C.
14. Carbon is dispersed in Na-K alloy. This results in swelling of graphite bearing materials such as cast iron.
15. Silicon dissolved in Na-K and alloyed with the steel bomb.
16. Silver dissolved in Na-K.
17. Platinum dissolved in Na-K when tested in a nickel bomb at 600°C. The platinum was suspended so as not to touch the bomb, yet it was found alloyed with the nickel bomb.

F. Current Program

1. Static Tests

- a. The static testing equipment has been revised to give several times the testing capacity with more flexibility and control of conditions of test.
- b. The current static testing program includes the following:
 - (1) Completion of tests in purified M.S.A. Na-K alloy of all materials which have been tested in impure home-made Na-K alloy.
 - (2) Testing of the following metals:
 - Type 347 stainless steel (Cb stabilized)
 - Type 316 stainless steel (Mo stabilized)
 - Type 310 stainless steel (25% Cr-20% Ni)
 - Nitrided steel
 - S.A.E. 52100 steel (ball bearing type)
 - Hadfield Mn steel
 - Hastelloy (Ni, Mo, Fe)
 - Stellite (Co, Cr)
 - Carboloy (W-C)
 - Be-Copper
- c. Determination of the effects of metals which are sure to be used in our work on the rate of attack of all metals tested.

2. Dynamic Tests

- a. Two thermal convection systems for dynamic tests are being made; one of low-carbon steel and another of commercially pure nickel. A third system will be made of type 347 stainless steel (Cb stabilized 18-8).
- b. A small dynamic system in which Na-K alloy will be circulated at high velocity by means of a centrifugal pump is now ready for use.

Table I. Materials Tested in Na-K Alloy and Approximate Chemical Compositions

Material	% C	% Mn	% S	% P	% Si	% Ni	% Cu	% Fe	% Cr	% Zn	% Sn	% Al
U	-	-	-	-	-	-	-	-	-	-	-	-
Th	-	-	-	-	-	-	-	-	-	-	-	-
Be	-	-	-	-	-	-	-	-	-	-	-	-
Cb (commercially pure)	-	-	-	-	-	-	-	-	-	-	-	-
Armco iron	.012	.017	.025	.005	-	-	-	Bal.	-	-	-	-
S.A.E. 1020 steel	.15-.25	.3-.6	.055 max.	.045 max.	-	-	-	Bal.	-	-	-	-
Ni (commercially pure)	.1	.2	.005	-	.05	99.4	.1	.15	-	-	-	-
Z-Ni	-	-	-	-	-	99.8	-	-	-	-	-	-
Invar	.18	.42	-	-	-	35.5	-	Bal.	-	-	-	-
Inconel	.08	.25	.015	-	.25	79.5	.2	6.5	13	-	-	-
Monel	-	-	-	-	-	65.3	28.4	0.37	-	-	-	-
80% Ni-20% Mn	-	20	-	-	-	80	-	-	-	-	-	-
Type 430 stainless	.12 max.	-	-	-	-	-	-	-	14-18	-	-	-
Type 302 stainless	.08-.2	-	-	-	-	8-9	-	-	17.5-19	-	-	-
Cu (commercially pure)	-	-	-	-	-	-	-	-	-	-	-	-
Al bronze	-	.1	-	-	.1	.1	Bal.	3.07	-	.1	.1	9.22
Sn bronze	-	-	-	-	-	-	Bal.	-	-	0.5	9.9	-
70% Cu-30% Mn	-	30	-	-	-	-	70	-	-	-	-	-
Graphite	100	-	-	-	-	-	-	-	-	-	-	-
Cast iron - Mechanite	3.0	1.4	.1	-	.8-1.0	-	-	Bal.	-	-	-	-
Si (commercially pure)	-	-	-	-	-	-	-	-	-	-	-	-
Ag (commercially pure)	-	-	-	-	-	-	-	-	-	-	-	-
Pt (commercially pure)	-	-	-	-	-	-	-	-	-	-	-	-

*Includes small amounts of Co.

Table II. Summary of Static and Dynamic Test Results in Na-K Alloy

Material	Bomb	Temp. (°C)	Time (days)	Rate of Weight Change (mg/cm ² /hr)	Remarks
U	low-C steel	600 to 800	1 to 3	-1 to -5	Heavy oxide film; loss due to flaking.
Th	"	600 to 800	1	-0.1 to -3	Specimen covered by thin black oxide.
U	"	*600	6	-0.009	Specimen covered by thin grey oxide.
Th	"	*600	6	+0.006	Specimen covered by thin grey oxide.
Be	"	800	3-1/2	-0.05	Specimen etched and covered with loose thin grey flakes.
"	"	800	30	-0.005	Specimen coated with thin grey film.
"	"	*600	6	-0.0015	Oxygen added to M.S.A. Na-K. Specimen coated with grey film.
"	"	600	6	+0.003	Specimens bright.
"	Dynamic	200 to 300	6	+0.0034	Black film.
"	Dynamic	500	6	-0.0124	
Cb	low-C steel	*600	6	-0.0016	Dull grey surface.
"	18-8	*600	6	-0.0064	Black film; not adherent; grey under film.
SAE 1020	Dynamic	500	6-1/2	-0.001	Specimens bright; decarburization up to 20 mil.
Armco Fe	Dynamic	500	13	-0.0001	Specimen bright.
Plated Radio Fe	Dynamic	500	5-1/2	-0.0007	Specimen bright.
SAE 1020	low-C steel	700	1 & 2	-0.02	Surface bright, carbide spe-
"	"	700	7	-0.01	roidization and surface decar-
"	"	800	8	-0.002	burization.
"	"	800	30	-0.001	Surface bright; no appreciable decarburization.
SAE 1020 (& 18-8)	18-8	400	9-1/2	-0.0001	Surface bright.
"	"	800	7-1/2	-0.08	Surface etched; Fe crystals on bomb wall.
Ni	"	*600	1	-0.0005	Slightly dulled by etch.
"	"	*600	6	-0.0001	Slightly dulled by etch.
"	low-C steel	600	5	+0.003	Thin grey film.
"	"	*600	6	+0.0007	Thin grey film.
"	Dynamic	500	6	nil	Specimens dull.
Z-Ni	low-C steel	600	7	+0.02	Heavy dark film.
Invar	"	600	6	+0.0007	No visible attack.
"	18-8	600	6	+0.0006	No visible attack.
"	low-C steel	800	6	+0.005	Specimen bright.
Inconel	"	300	30	nil	No visible attack.
"	"	600	5	+0.01	Dull grey film.
"	18-8	600	6	+0.003	Dull grey film.
"	low-C steel	800	6	+0.1	Mottled grey film.
Monel	"	300	30	nil	No visible attack.
"	"	600	7	+0.02	0.5 mil Fe-Ni Film.
"	18-8	600	6	-0.001	Lightly etched surface.
80% Ni-20% Mn	"	600	5	+0.002	Surface bright.
17% Cr Steel	low-C steel	800	7	-0.07	Dull green film; Fe crystals on bomb wall.
18-8 Stainless	"	400	9-1/2	+0.0008	Very thin film overlying Cr oxide.
"	"	800	7-1/2	+0.06	Dull grey film overlying Cr oxide. Fe crystals on bomb wall.
Cu	"	800	4	-2.5	Strongly attacked; Cu crystals on bomb wall.
Al Bronze	"	800	3	-0.015	Dull etched surface.
Sn Bronze	"	800	8	-0.9	Tin phase etched out.
70% Cu-30% Mn	"	600	6	Dissolved	5 mil sheet weighing 1.2 grams completely dissolved.
Graphite	"	Room	--	Dispersed	5 grams graphite dispersed in 10 grams Na-K.
Cast Fe	"	800	1	+0.75	Swelled & distorted; impregnated with Na-K.
Si	"	800	6	Dissolved	Formed thick shell containing 12% Si on I.D. of bomb.
Ag	"	800	1-1/2	Dissolved	10 gram sample completely dissolved.
Pt	Ni	600	1	Dissolved	Pt not in contact with Ni bomb but alloyed with it.

*M.S.A. Na-K deoxidized with uranium turnings. All other tests made with relatively impure Na-K alloy made from sticks of Na and K.

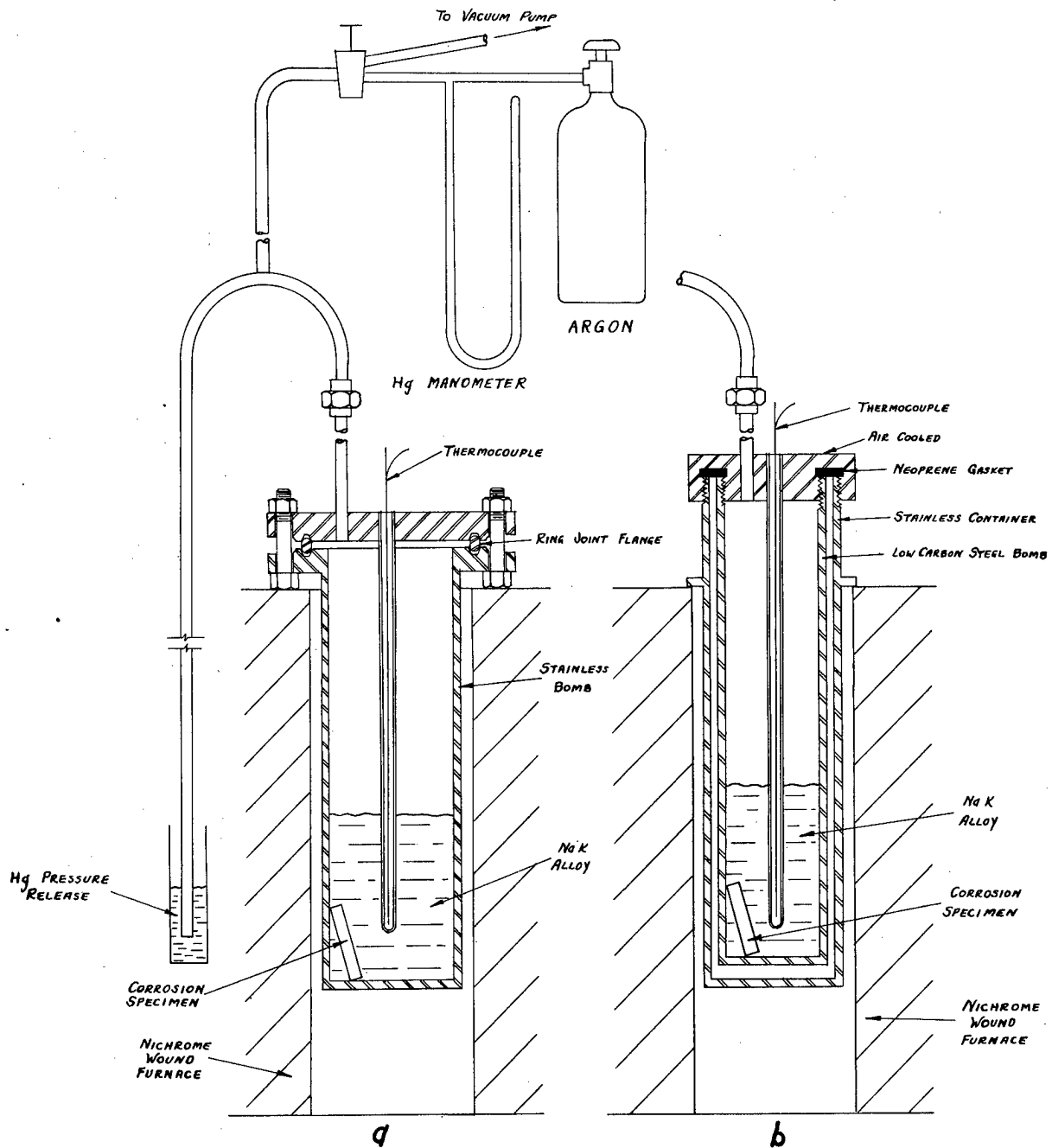


FIG 1
 ONE - HALF SIZE
STATIC NaK CORROSION TESTING UNITS

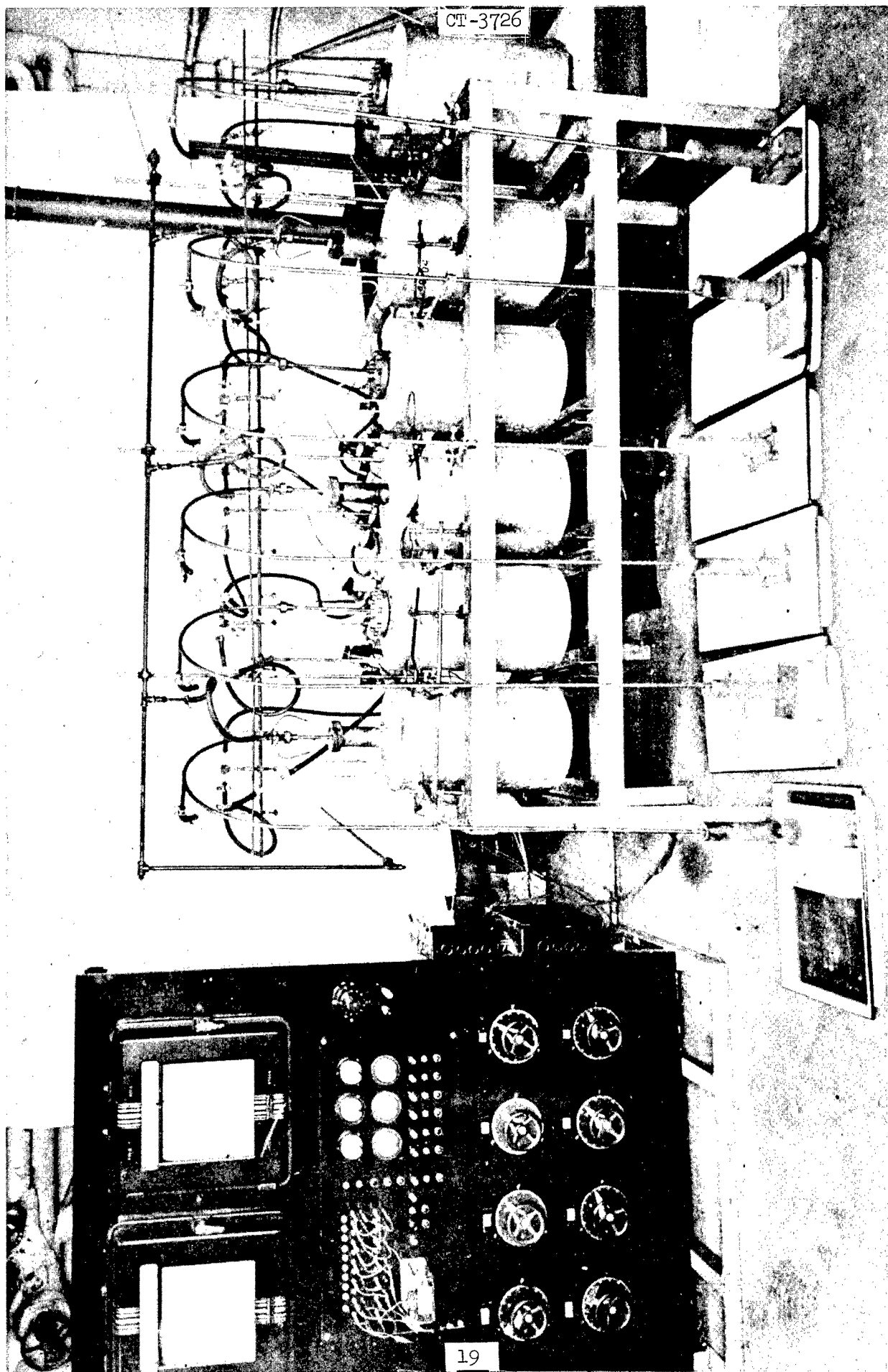


Fig.2 - Photograph of 6 static Na-K units and control panel.

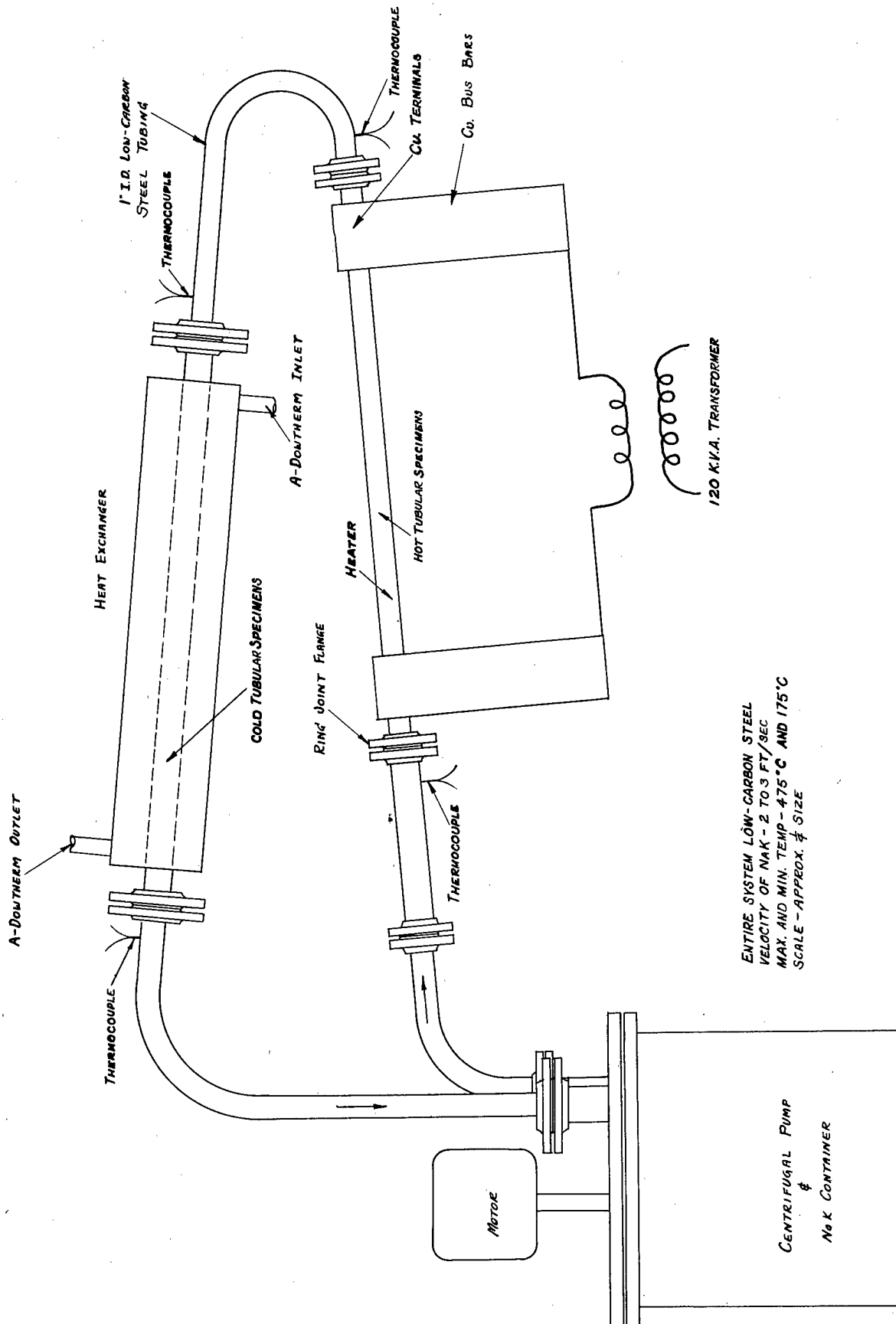


FIG 3
LARGE DYNAMIC NAK CORROSION SYSTEM

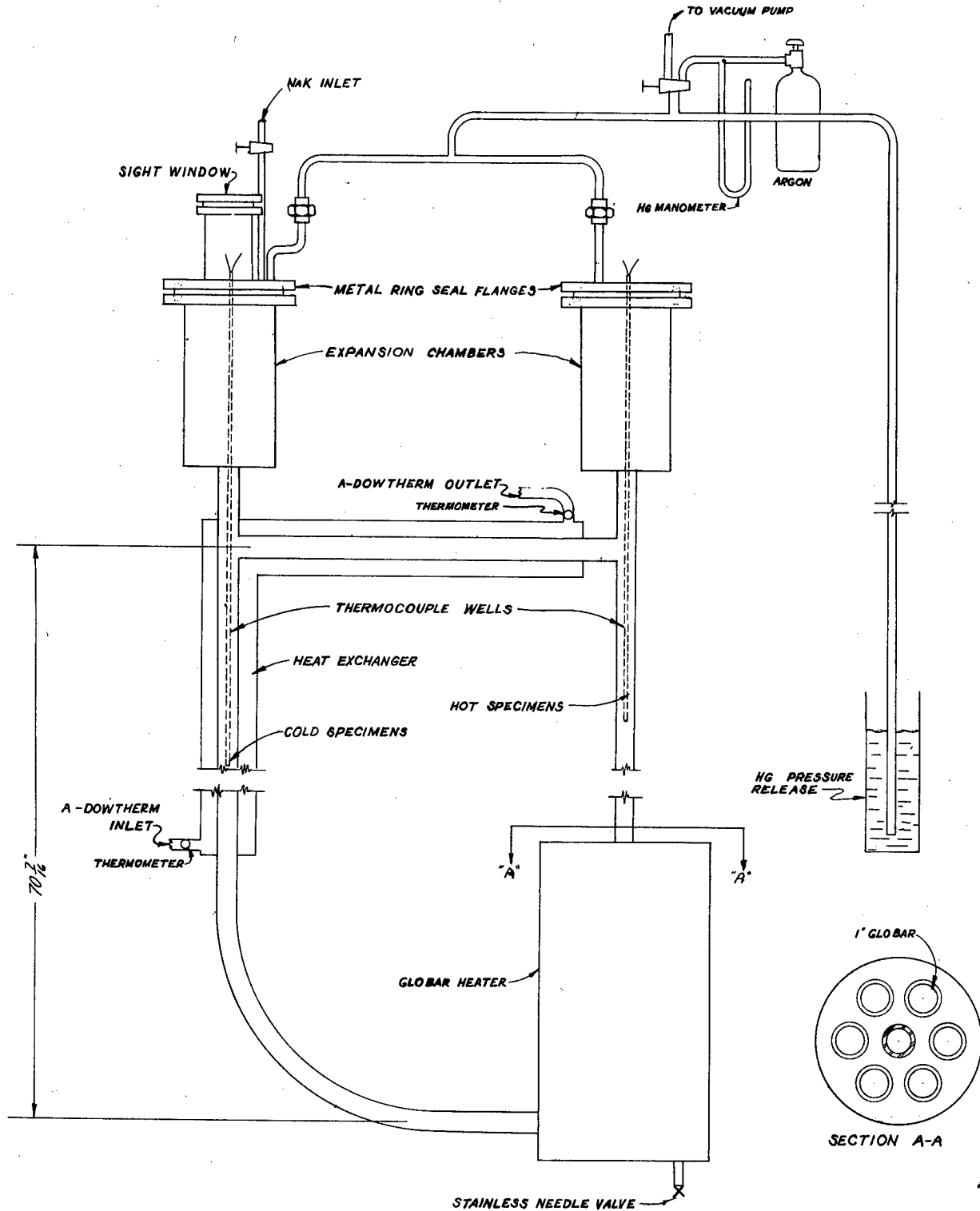
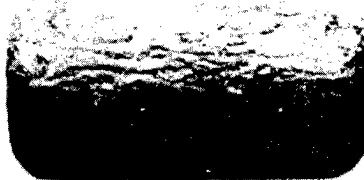


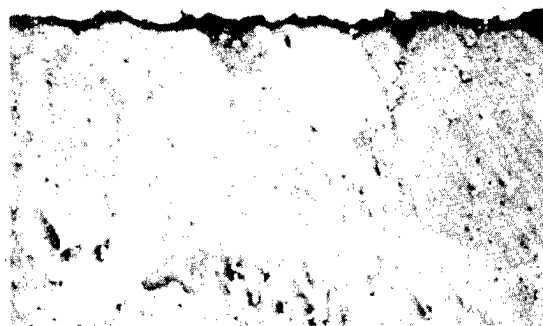
FIG. 4
THERMAL CONVECTION NaK CORROSION SYSTEM



M-3072

2-X

Fig. 5 - Appearance of uranium specimen after testing for 3 days at 700°C in Na-K alloy contaminated with oxygen. The rounded corners are due to flaking off of heavy oxide layers during test with resulting rate of weight loss of $-5 \text{ mg/cm}^2/\text{hr}$.



4410 Unetched-Polarized Light 75-X

Fig. 6 - Microscopic section through oxidized surface of uranium specimen shown in Fig. 5. Note that attack is transcrystalline rather than intergranular and that inclusions provided a further avenue of attack.

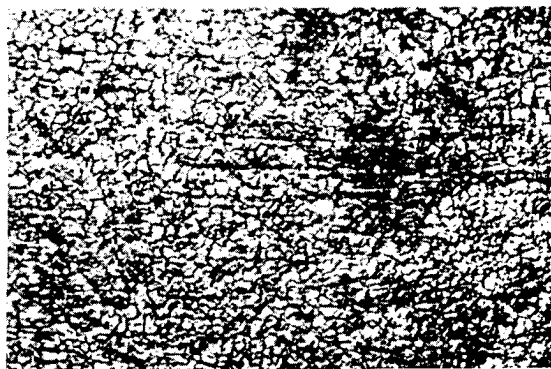


4404

Unetched

100-X

Fig. 7 - Section through corroded surface of thorium specimen after testing for 1 day at 800°C in Na-K alloy contaminated with oxygen. The attack was transcrystalline and the oxide layer spalled off during test resulting in a rate of weight loss of $-3 \text{ mg/cm}^2/\text{hr}$.



M-3071

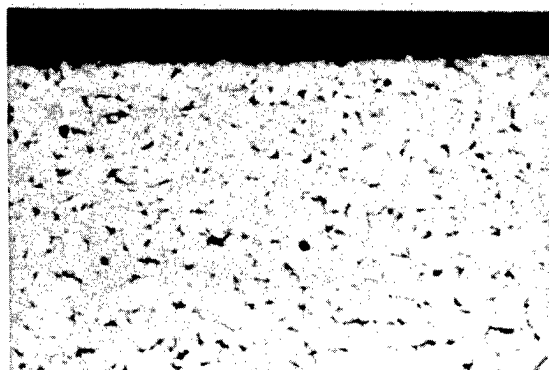
10-X

Fig. 8 - Appearance of beryllium specimen surface after 30 days at 800°C showing grain outlines developed by attack of Na-K alloy. The rate of weight loss was $-0.005 \text{ mg/cm}^2/\text{hr}$.

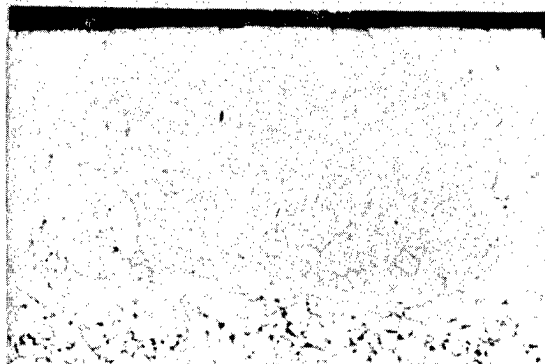


4405 Unetched-Polarized Light 75-X

Fig. 9 - Section through surface of beryllium specimen shown in Fig. 8 showing limited extent of intergranular attack.



4253 Nital etch 100-X



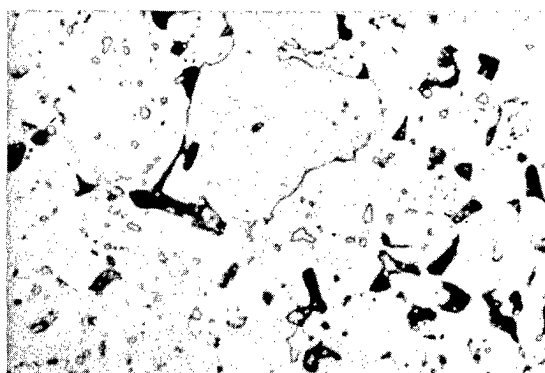
4255 Nital etch 100-X

Figs.10 & 11 - Typical structure of S.A.E. 1020 steel before and after dynamic tests in Na-K alloy at approximately 500°C showing extensive decarburization and grain growth. The rate of weight loss was $-0.001 \text{ mg/cm}^2/\text{hr}$.



4213 Nital etch 500-X

Fig.12 - Structure of S.A.E. 1020 steel before testing at 700°C and 800°C. The surfaces of the specimens were free from decarburization before test.



4212 Nital etch 500-X

Fig.13 - S.A.E. 1020 steel after 7 days at 700°C (slightly below the eutectoid temperature). Note extensive spheroidization of carbides. The surface was totally decarburized to a depth of 10 mils and partially decarburized to 25 mils. The rate of weight loss was $-0.01 \text{ mg/cm}^2/\text{hr}$.



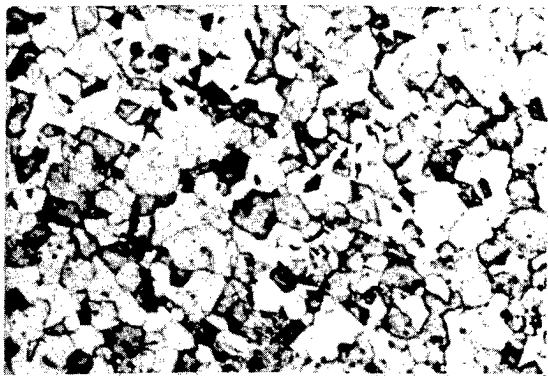
4209 Nital etch 500-X

Fig.14 - S.A.E. 1020 steel after 8 days at 800°C which is above the eutectoid temperature so that carbides were in solution and their mode of occurrence was a function of rate of cooling only. The temperature was not high enough to completely dissolve the massive ferrite. There was practically no surface decarburization, and the rate of weight loss was $-0.002 \text{ mg/cm}^2/\text{hour}$.



M-4000 10-X

Fig.15 - Iron crystals deposited on wall of static test bomb at the Na-K alloy level when 18-8 stainless steel was tested in a low-carbon steel bomb at 800°C.



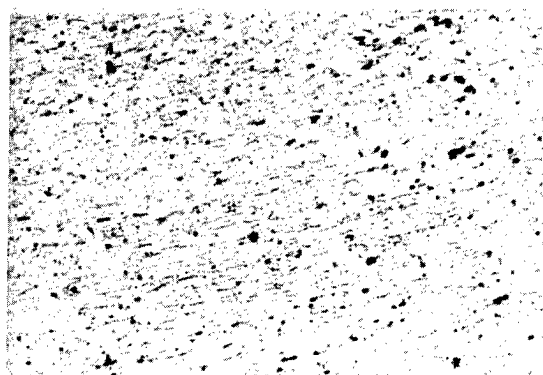
CT-3726



4335 HNO_3 -Acetic etch 100-X

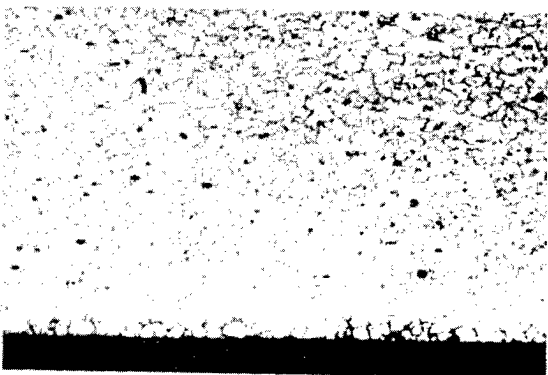
4345 HNO_3 -Acetic etch 150-X

Figs.16 & 17 - Typical microstructure of commercially pure nickel before and after testing in Na-K alloy at elevated temperatures in a low-carbon steel bomb. Note the extensive grain growth and the thin continuous surface film. Considerable evidence shows this film to be a solid solution of iron and nickel.



4356 HNO_3 -Acetic etch 250-X

Fig.18 - Rolled structure of Invar before testing in Na-K alloy.



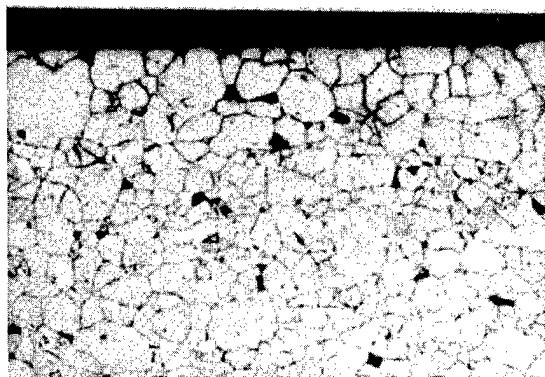
4354 HNO_3 -Acetic etch 250-X

Fig.19 - Invar after 6 days at 600°C showing recrystallization and incipient intergranular corrosion. The specimen gained weight at the rate of $+0.007 \text{ mg/cm}^2/\text{hr}$.

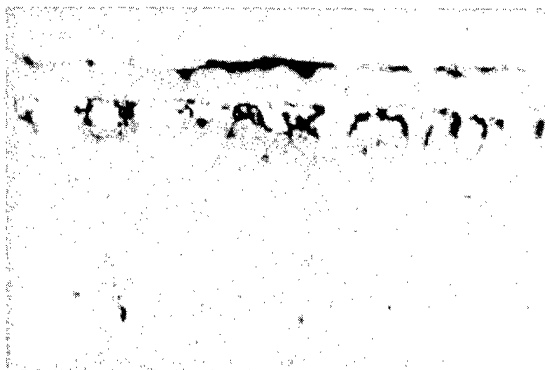


4369 HNO_3 -Acetic etch 250-X

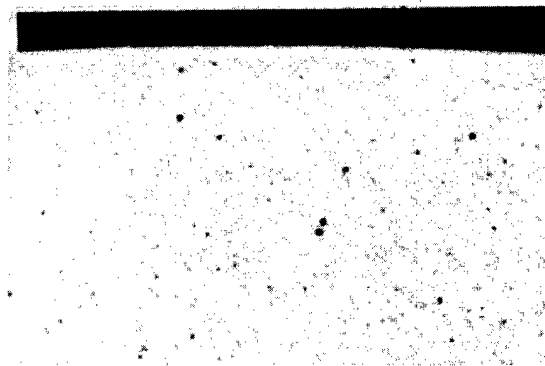
Fig. 20 - Invar after 6 days at 800°C showing extensive grain growth and incipient intergranular corrosion. The specimen gained weight at the rate of $+0.005 \text{ mg/cm}^2/\text{hr}$.



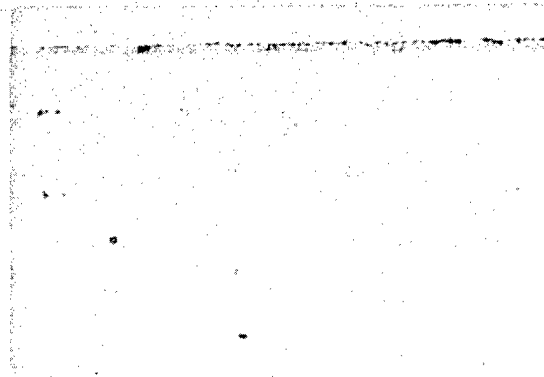
4416 HNO₃-Acetic etch 250-X
Fig.21 - As-received structure of Inconel.



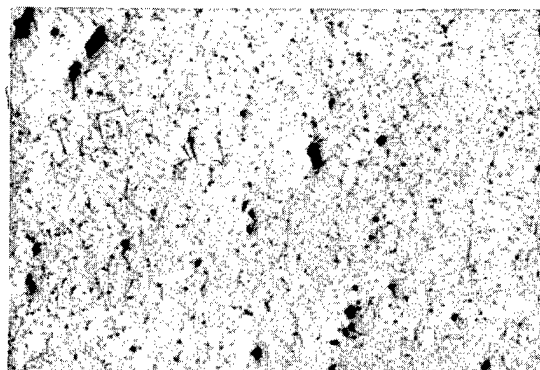
4423 Unetched 250-X
Fig.23 - Inconel after 6 days at 800°C showing agglomeration of precipitated particles and fingered layer of chromium oxide as a result of intergranular attack. The surface layer is similar to the iron-nickel films found when all high nickel alloys are tested in the presence of iron. The specimen gained weight at the rate of +0.1 mg/cm²/hour.



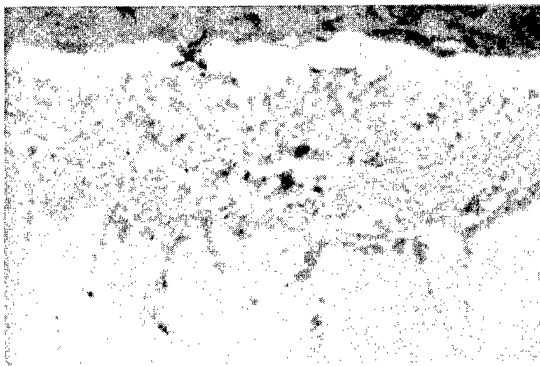
4427 FeCl₃ & HCl etch 100-X
Fig.25 - 18-8 stainless steel after 9½ days at 400°C in a low-carbon steel bomb. There appeared to be a thin surface film on this specimen, and it showed incipient intergranular corrosion. It gained only +0.0008 mg/cm²/hour.



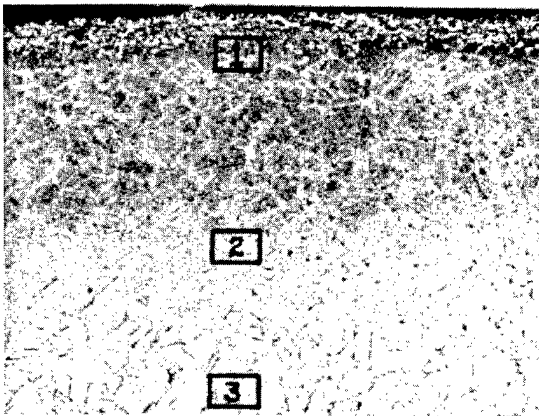
4421 Unetched 250-X
Fig.22 - Inconel after 5 days at 600°C showing no appreciable grain growth. There are traces of chromium oxide under a thin iron-nickel film. The specimen gained weight at the rate of +0.01 mg/cm²/hour.



4412 Chromic-Elect.etch-oblique 100-X
Fig.24 - Structure of type 302 (18-8) austenitic stainless steel before Na-K corrosion test. Oblique illumination was needed to bring out grain structure.



4215 Unetched 250-X
Fig.26 - Surface microstructure of 18-8 stainless steel after 7½ days at 800°C showing the intergranular corrosion product (chromium oxide) underlying the iron-nickel layer deposited on the surface. Identity of these features was established by chemical analysis. The specimen gained +0.06 mg/cm²/hour.



4411 Chromic-elect.etch 100-X

Fig.27-General view of specimen from figure 26 showing transition zones from austenitic (bottom) to ferritic (top) phases. Boxes show general location of high magnification pictures shown in figures 28, 29, and 30.

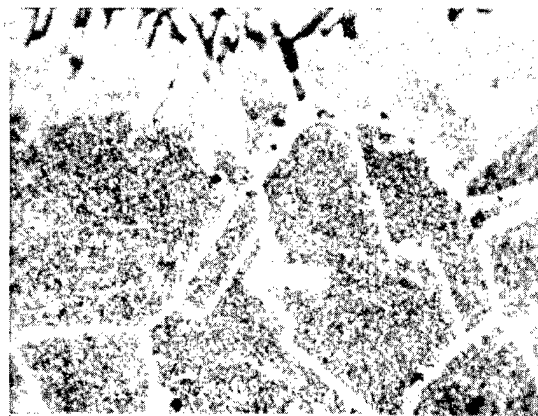
4407 $\text{FeCl}_3 + \text{HCl}$ etch 1000-X

Fig.28 - Area #1 of figure 27 showing intergranular progress of corrosion product from surface (top) inward. Structure at this point is magnetic indicating transformation of matrix to ferrite. Note abundance of boundary and cleavage plane constituent.

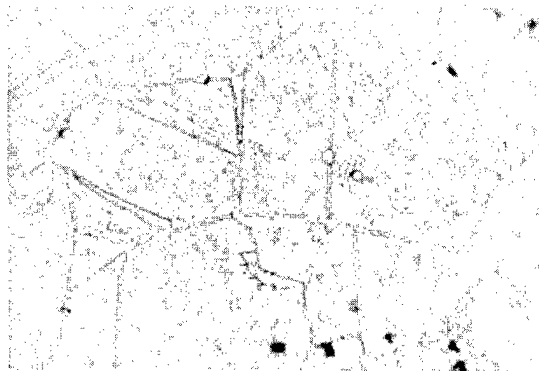
4408 $\text{FeCl}_3 + \text{HCl}$ etch 1000-X

Fig.29 - Area #2 of figure 27 showing intermediate structure with heavily precipitated constituent migrating to the grain boundaries.

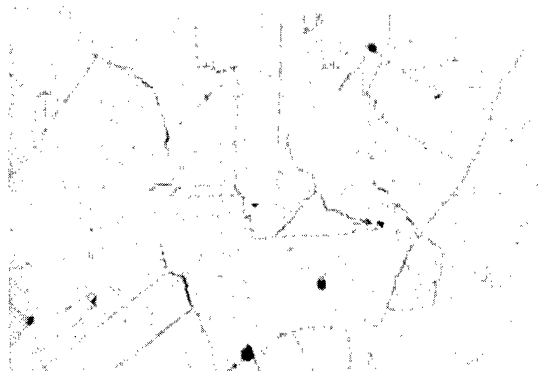
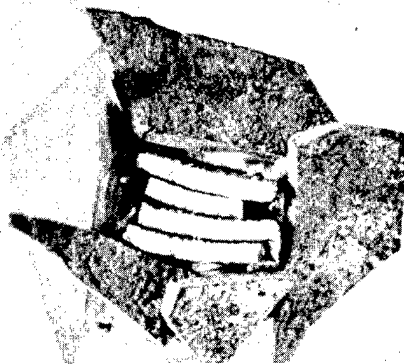
4409 $\text{FeCl}_3 + \text{HCl}$ etch 1000-X

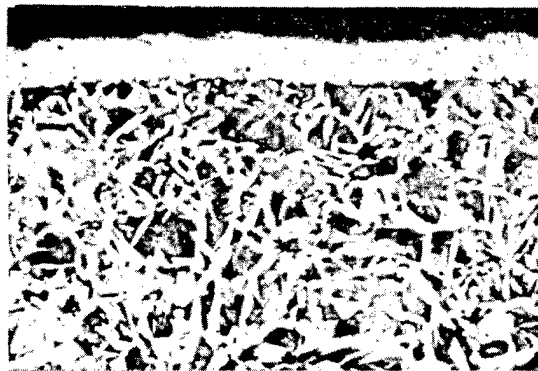
Fig.30 - Area #3 of figure 27 showing incipient precipitation within grains at center of specimen and no appreciable grain boundary separation.



M-4003

2-X

Figs.31 & 32 - Thick layer formed on bomb wall when silicon was tested in a low-carbon steel bomb. The layer contained 12% Si and balance Fe. It was very brittle and cleaved from the bomb wall on cooling. Fig.32 shows the progress of the formation of the Fe-Si alloy on the inner surface of the bomb.



4421

Nital etch

100-X